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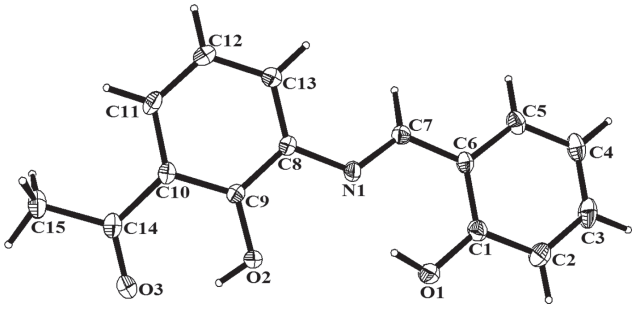
Crystal structure of (E)-1-(2-hydroxy-3-{[2-hydroxybenzylidene]amino}phenyl)ethan-1-one, $C_{15}H_{13}NO_3$



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Abstract

$C_{15}H_{13}NO_3$, monoclinic, $P2_1/c$ (no. 14) $a = 7.5158(6)$ Å, $b = 6.3940(5)$ Å, $c = 25.881(3)$ Å, $\beta = 93.243(8)°$, $Z = 4$, $V = 1241.74(19)$ Å^3, $R_{gt}(F) = 0.0547$, $wR_{ref}(F^2) = 0.1582$, $T = 173$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were used as received. A solution of salicylaldehyde (810 mg, 6.62 mmol) in ethanol (25 mL) was added to the ethanolic solution (25 mL) of 3-amino-2-hydroxyacetophenone (1000 mg, 6.62 mmol). The solution was stirred with reflux at 328 K for 12 h, after which the mixture was filtered and the filtrate was washed successively with ethanol and hexane in the ratio 1:4, respectively. Then the product was dried using a vacuum desiccator on calcium chloride which resulted in a clear light brown powder

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Table 1: Data collection and handling.

Crystal:	Clear light yellow block
Size:	0.24 × 0.23 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	26.0°, 99.7%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4274, 2430, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1626
$N(param)_{refined}$:	175
Programs:	CrysAlis^{PRO} [20], SHELX [19]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

	x	y	z	U_{iso}*/U_{eq}
O1	0.1617(2)	0.6715(3)	0.33013(6)	0.0451(5)
H1	0.2499	0.6836	0.3501	0.068*
O2	0.38250(18)	0.7432(2)	0.45362(5)	0.0300(4)
H2A	0.3615	0.7483	0.4843	0.045*
O3	0.4504(2)	0.7575(2)	0.55070(6)	0.0347(4)
N1	0.4910(2)	0.7272(3)	0.35802(7)	0.0264(4)
C1	0.2100(3)	0.6927(4)	0.28098(8)	0.0323(5)
C2	0.0808(3)	0.6716(4)	0.24065(9)	0.0416(6)
H2	−0.0363	0.6420	0.2478	0.050*
C3	0.1258(3)	0.6944(4)	0.19022(9)	0.0446(7)
H3	0.0378	0.6805	0.1637	0.053*
C4	0.2986(3)	0.7375(3)	0.17807(9)	0.0404(6)
H4	0.3271	0.7530	0.1438	0.048*
C5	0.4278(3)	0.7569(3)	0.21762(8)	0.0330(5)
H5	0.5446	0.7848	0.2097	0.040*
C6	0.3872(3)	0.7357(3)	0.26959(8)	0.0261(5)
C7	0.5255(3)	0.7525(3)	0.31029(8)	0.0269(5)
H7	0.6416	0.7821	0.3019	0.032*
C8	0.6227(3)	0.7349(3)	0.39886(8)	0.0246(5)
C9	0.5603(3)	0.7423(3)	0.44895(8)	0.0241(5)
C10	0.6816(3)	0.7503(3)	0.49268(8)	0.0259(5)
C11	0.8643(3)	0.7468(3)	0.48478(9)	0.0332(6)
H11	0.9458	0.7529	0.5131	0.040*
C12	0.9252(3)	0.7344(3)	0.43580(9)	0.0365(6)
H12	1.0470	0.7299	0.4313	0.044*
C13	0.8050(3)	0.7286(3)	0.39309(9)	0.0316(5)
H13	0.8474	0.7203	0.3601	0.038*
C14	0.6119(3)	0.7611(3)	0.54474(8)	0.0298(5)
C15	0.7390(3)	0.7770(4)	0.59150(9)	0.0413(6)
H15A	0.8114	0.6534	0.5941	0.062*
H15B	0.6728	0.7904	0.6220	0.062*
H15C	0.8139	0.8974	0.5883	0.062*

(yield 73.4%, m.p. 393 K). Several single crystals suitable for X-ray crystallographic analysis were obtained by purifying the compound using the method of column chromatography with appropriate solvents at room temperature. Elemental analysis: Anal. calcd for $C_{15}H_{13}NO_3$: C, 70.58%; H, 5.13%; N, 5.49%; Found: C, 70.56%; H, 5.09%; N, 5.52%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Generally, Schiff bases are synthesized through the condensation reaction of aldehydes or ketones with primary amines [1–5]. It is no gainsaying that the application of Schiff base compounds and their derivatives have become indispensable in the development of metal complexes in modern coordination chemistry [6–8], the classical catalytical processes [9], biological activities [10], photochemical [11, 12] and magnetic materials [13] and supramolecular architectures [14, 15].

In the crystal structure of the title compound bond distances and angles are in the normal ranges. There are two intramolecular O1-H1···N1 and O2-H2A···O3 hydrogen bonding interactions, which are in accord with the literature [16–18].

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